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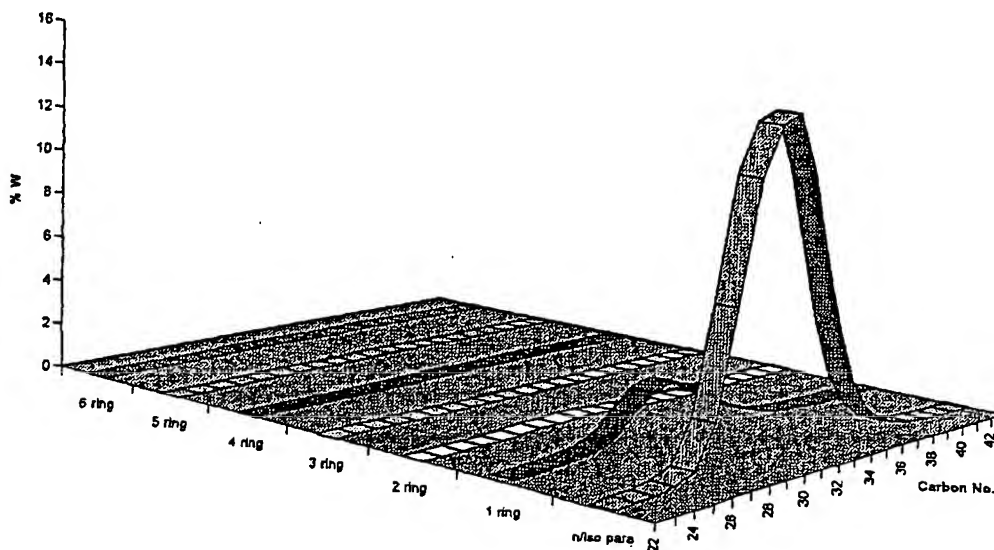
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(54) Title: LUBRICANT COMPOSITION



(57) Abstract: A lubricant composition comprising a base oil and one or more additives wherein the lubricant composition has a kinematic viscosity at 100 °C of more than 5.6 cSt, a cold cranking simulated dynamic viscosity at -35 °C according to ASTM D 5293 of less than 62 centiPoise (cP) and a mini rotary viscosity test value of less than 60000 cP according to ASTM d 4684, wherein the base oil has been obtained from a waxy paraffinic Fischer-Tropsch synthesized hydrocarbons.

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## LUBRICANT COMPOSITION

The invention is directed to a lubricant composition comprising a base oil and one or more additives wherein the lubricant composition has a kinematic viscosity at 100 °C of more than 5.6 cSt, a cold cranking simulated dynamic viscosity at -35 °C according to ASTM D 5293 of less than 62 centiPoise (cP) and a mini rotary viscosity test value of less than 60000 cP according to ASTM D 4684.

Such lubricant compositions are also referred to as SAE 0W-x compositions. SAE stands for Society of Automotive Engineers in the USA. The "0" number in such a designation is associated with a maximum viscosity requirement at -35 °C for that composition as measured typically by a cold cranking simulator (VdCCS) under high shear. The second number "x" is associated with a kinematic viscosity requirement at 100 °C.

The minimum high temperature viscosity requirement at 100 °C is intended to prevent the oil from thinning out too much during engine operation which can lead to excessive wear and increased oil consumption. The maximum low temperature viscosity requirement, VdCCS, is intended to facilitate engine starting or cranking in cold weather. To ensure pumpability the cold oil should readily flow or slump into the well for the oil pump, otherwise the engine can be damaged due to insufficient lubrication. The mini rotary viscosity (MRV) requirement is intended to ensure a minimum pumpability performance.

US-A-5693598 describes a lubricant formulation according to 0W-20 based on so-called poly-alpha olefins. Poly-alpha olefins are prepared by oligomerisation of alpha olefins (PAO) as for example described in

hydroisomerisation step and a pour point reducing step, wherein said hydroisomerisation step and pour point reducing step are performed as:

- 5 (a) hydrocracking/hydroisomerising a Fischer-Tropsch product,
- (b) separating the product of step (a) into at least one or more fuel fractions and a base oil precursor fraction, and
- 10 (c) performing a pour point reducing step to the base oil precursor fraction obtained in step (b).

Examples of Fischer-Tropsch synthesis processes steps to prepare said Fischer-Tropsch product and hydroisomerisation steps (a) are known from the so-called commercial Sasol process, the commercial Shell Middle

15 Distillate Process or the non-commercial Exxon process.

The Fischer-Tropsch product used in step (a) will contain no or very little sulphur and nitrogen containing compounds. This is typical for a product derived from a Fischer-Tropsch reaction which uses synthesis gas

20 containing almost no such impurities. Sulphur and nitrogen levels will generally be below the detection limit, which is currently 1 ppm for nitrogen and 5 ppm for sulphur.

The Fischer-Tropsch product may optionally be

25 subjected to a mild hydrotreatment step in order to remove any oxygenates and saturate any olefinic compounds present in the reaction product of the Fischer-Tropsch reaction. Such a hydrotreatment is described in EP-B-668342. The mildness of the hydrotreating step is

30 preferably expressed in that the degree of conversion in this step is less than 20 wt% and more preferably less than 10 wt%. The conversion is here defined as the weight percentage of the feed boiling above 370 °C, which reacts to a fraction boiling below 370 °C.

moiety, in particular fluorine, or a phosphorous moiety to the carrier, may enhance the acidity of the catalyst carrier. Examples of suitable hydrocracking/hydro-isomerisation processes and suitable catalysts are described in WO-A-0014179, EP-A-532118, EP-A-666894 and the earlier referred to EP-A-776959.

Preferred hydrogenation/dehydrogenation functionalities are Group VIII noble metals, for example palladium and more preferably platinum. The catalyst may comprise the hydrogenation/dehydrogenation active component in an amount of from 0.005 to 5 parts by weight, preferably from 0.02 to 2 parts by weight, per 100 parts by weight of carrier material. A particularly preferred catalyst for use in the hydroconversion stage comprises platinum in an amount in the range of from 0.05 to 2 parts by weight, more preferably from 0.1 to 1 parts by weight, per 100 parts by weight of carrier material. The catalyst may also comprise a binder to enhance the strength of the catalyst. The binder can be non-acidic. Examples are clays and other binders known to one skilled in the art.

In step (a) the feed is contacted with hydrogen in the presence of the catalyst at elevated temperature and pressure. The temperatures typically will be in the range of from 175 to 380 °C, preferably higher than 250 °C and more preferably from 300 to 370 °C. The pressure will typically be in the range of from 10 to 250 bar and preferably between 20 and 80 bar. Hydrogen may be supplied at a gas hourly space velocity of from 100 to 10000 Nl/l/hr, preferably from 500 to 5000 Nl/l/hr. The hydrocarbon feed may be provided at a weight hourly space velocity of from 0.1 to 5 kg/l/hr, preferably higher than 0.5 kg/l/hr and more preferably lower than 2 kg/l/hr. The ratio of hydrogen to

fraction is obtained boiling in the specified range and having a kinematic viscosity, which relates to the base oil end product(s) specification. The kinematic viscosity at 100 °C of the base oil precursor fraction is preferably between 3 and 10 cSt.

Suitably the above described waxy paraffinic product or base oil precursor fraction is obtained in the hydroisomerisation process step wherein the content on non-cyclic iso-paraffin compounds, relative to the total of non-cyclic iso- and normal paraffins, is increased to above 90 wt%. This waxy paraffinic product, which boils for the greater part above 370 °C, is subsequently subjected to a pour point reducing step. The pour point reducing step can be by means of solvent dewaxing or catalytic dewaxing according to the aforementioned publications. The dewaxed product is further purified in order to remove both a light and optionally a heavy fraction, such to obtain the base oil suitable for use in the lubricant formulation of the present invention.

Preferably the base oil is prepared by a process wherein the pour point reducing step is performed by means of a catalytic dewaxing. With such a process it has been found that base oils have a sufficiently low pour point of, for example as low as -40 °C. The catalytic dewaxing process can be performed by any process wherein in the presence of a catalyst and hydrogen the pour point of the base oil precursor fraction is reduced as specified above. Suitable dewaxing catalysts are heterogeneous catalysts comprising a molecular sieve and optionally in combination with a metal having a hydrogenation function, such as the Group VIII metals. Molecular sieves, and more suitably intermediate pore size zeolites, have shown a good catalytic ability to reduce the pour point of the base oil precursor fraction under catalytic dewaxing conditions. Preferably the

A preferred class of dewaxing catalysts comprise intermediate zeolite crystallites as described above and a low acidity refractory oxide binder material which is essentially free of alumina as described above, wherein  
5 the surface of the aluminosilicate zeolite crystallites has been modified by subjecting the aluminosilicate zeolite crystallites to a surface dealumination treatment. A preferred dealumination treatment is by contacting an extrudate of the binder and the zeolite  
10 with an aqueous solution of a fluorosilicate salt as described in for example US-A-5157191 or WO-A-0029511. Examples of suitable dewaxing catalysts as described above are silica bound and dealuminated Pt/ZSM-5, silica bound and dealuminated Pt/ZSM-23, silica bound and  
15 dealuminated Pt/ZSM-12, silica bound and dealuminated Pt/ZSM-22, as for example described in WO-A-0029511 and EP-B-832171.

Catalytic dewaxing conditions are known in the art and typically involve operating temperatures in the range  
20 of from 200 to 500 °C, suitably from 250 to 400 °C, hydrogen pressures in the range of from 10 to 200 bar, preferably from 40 to 70 bar, weight hourly space velocities (WHSV) in the range of from 0.1 to 10 kg of oil per litre of catalyst per hour (kg/l/hr), suitably  
25 from 0.2 to 5 kg/l/hr, more suitably from 0.5 to 3 kg/l/hr and hydrogen to oil ratios in the range of from 100 to 2,000 litres of hydrogen per litre of oil. By varying the temperature between 275 °C, suitably between 315 °C and 375 °C at between 40-70 bars, in the catalytic  
30 dewaxing step it is possible to prepare base oils having different pour point specifications varying from suitably -10 to -60 °C.

The lubricant composition suitably comprises between 65 and 85 wt% of the Fischer-Tropsch derived base oil.  
35 The remaining part of the composition consists of one or

desorption/Field Ionisation (FD/FI) interface, wherein FI (a "soft" ionisation technique) is used for the semi-quantitative determination of hydrocarbon types in terms of carbon number and hydrogen deficiency. The type

5 classification of compounds in mass spectrometry is determined by the characteristic ions formed and is normally classified by "z number". This is given by the general formula for all hydrocarbon species:  $C_nH_{2n+z}$ .

Because the saturates phase is analysed separately from

10 the aromatic phase it is possible to determine the content of the different (cyclo)-paraffins having the same stoichiometry. The results of the mass spectrometer are processed using commercial software (poly 32; available from Sierra Analytics LLC, 3453 Dragoo Park

15 Drive, Modesto, California 95350 USA) to determine the relative proportions of each hydrocarbon type and the average molecular weight and polydispersity of the saturates and aromatics fractions.

The base oil composition preferably has a content of

20 aromatic hydrocarbon compounds of less than 1 wt%, more preferably less than 0.5 wt% and most preferably less than 0.1 wt%, a sulphur content of less than 20 ppm and a nitrogen content of less than 20 ppm. The pour point of the base oil is preferably less than -30 °C and more

25 preferably lower than -40 °C. The viscosity index is preferably higher than 120. It has been found that the novel base oils typically have a viscosity index of below 140. The kinematic viscosity at 100 °C of the base oil is preferably between 4.0 and 8 cSt and the Noack volatility

30 is preferably lower than 14 wt%.

The above base oil is believed to be novel. Relevant publications like WO-A-0014188, WO-A-14187 and

WO-A-0014179 disclose base oils derived from a Fischer-Tropsch synthesis product containing more than 95 wt%

35 iso-paraffins. WO-A-0118156 describes a base oil derived

ratio of compounds having at least 60 or more carbon atoms and compounds having at least 30 carbon atoms of the Fischer-Tropsch product is at least 0.2, preferably at least 0.4 and more preferably at least 0.55.

5 Preferably the Fischer-Tropsch product comprises a  $C_{20}^{+}$  fraction having an ASF-alpha value (Anderson-Schulz-Flory chain growth factor) of at least 0.925, preferably at least 0.935, more preferably at least 0.945, even more preferably at least 0.955. The initial boiling point of  
10 the Fischer-Tropsch product may range up to 400 °C, but is preferably below 200 °C.

Such a Fischer-Tropsch product can be obtained by any process, which yields a relatively heavy Fischer-Tropsch product. Not all Fischer-Tropsch processes yield such a  
15 heavy product. Examples of suitable Fischer-Tropsch processes are described in WO-A-9934917 and in AU-A-698392. These processes may yield a Fischer-Tropsch product as described above.

The base oil as obtainable by the above processes has  
20 a pour point of less than -39 °C and a kinematic viscosity at 100 °C which is suitably between 4 and 8 cSt. The actual kinematic viscosity at 100 °C will depend on the specific OW-x grade one wishes to prepare. For the OW-20 and OW-30 lubricant grades a base oil  
25 having a kinematic viscosity at 100 °C of between 3.8 and 5.5 cSt is suitably used. For an OW-40 grade a base oil having a kinematic viscosity at 100 °C of between 5.5 and 8 cSt is suitably used.

The lubricant composition comprises one or more  
30 additives. Examples of additive types which may form part of the composition are dispersants, detergents, viscosity modifying polymers, extreme pressure/antiwear additives, antioxidants, pour point depressants, emulsifiers, demulsifiers, corrosion inhibitors, rust inhibitors,  
35 antistaining additives, friction modifiers. Specific



about 60 wt% C<sub>30</sub>+ product. The ratio C<sub>60</sub>+/C<sub>30</sub>+ was about 0.55.

Table 1

Recovered (wt%)	Temperature (°C)
Initial boiling point	82
10	249
30	424
50	553
70	671
90	>750

5 The Fischer-Tropsch product as thus obtained was continuously fed to a hydrocracking step (step (a)). In the hydrocracking step the Fischer-Tropsch product and a recycle stream consisting of the 370 °C<sup>+</sup> fraction of the effluent of step (a) was contacted with a hydrocracking catalyst of Example 1 of EP-A-532118 at a reactor temperature of 330 °C. The Fischer-Tropsch product WHSV  
10 was contacted at 0.8 kg/l.h and the recycle stream was contacted at 0.2 kg/l.h at a total pressure of 35 bar and a hydrogen partial pressure of 33 bar. The recycle gas rate was 2000 Nl/kg of total feed. The conversion of compounds boiling above 370 °C in the total feed which  
15 were converted to products boiling below 370 °C was 55 wt%. The product of the hydrocracking step was distilled into one or more fuels fractions boiling in the naphtha, kerosene and gas oil range and a bottom product boiling above 370 °C.

20 The 370 °C<sup>+</sup> fraction thus obtained was in turn distilled in a vacuum distillation column, wherein the feed rate to the column was 750 g/h, the pressure at the

Table 3

Density d20/4	818
Mean boiling point (50 wt% recovered)	448 °C
Kinematic viscosity at 40 °C	23.4 cSt
Kinematic viscosity at 100 °C	4.9 cSt
Viscosity index	128
Pour point	-55 °C
Noack volatility	6.8 wt%

Example 3

74.6 weight parts of a base oil, having the properties as listed in Table 4 and which was obtained by catalytic dewaxing of a hydroisomerised/hydrocracked Fischer-Tropsch product as illustrated by Examples 1 and 2, was blended with 14.6 weight parts of a standard detergent inhibitor additive package, 0.25 weight parts of a corrosion inhibitor and 10.56 weight parts of a viscosity modifier. The properties of the resulting composition are listed in Table 5. Table 5 also shows the OW-30 specifications for motor gasoline lubricants. It is clear that the composition as obtained in this Example meets the requirements of an OW30 motor gasoline specification.

Comparative experiment A

54.65 weight parts of a poly-alpha olefin-4 (PAO-4) and 19.94 weight parts of a poly-alpha olefin-5 (PAO-5), having the properties as listed in Table 1 were blended with the same quantity and quality of additives as in Example 3. The properties of the resulting composition are listed in Table 5. This experiment and Example 3 shows that a base oil as obtained by the present invention can be successfully used to formulate OW-30 motor gasoline lubricants using the same additives as

and is measured according to ASTM D 5293, (6) MRV cP @ -40 °C stands for mini rotary viscometer test and is measured according to ASTM D 4684, (7) pour point according to ASTM D 97, (8) Noack volatility as  
 5 determined by ASTM D 5800.

Table 5

	OW-30 specifi- cations	Example 3	Comparative experiment A
kinematic viscosity at 100 °C (cSt)	9.3-12.5	9.69	9.77
VDCCS P @ -35 °C (cP)	62.0 max	61.2	48.3
MRV cP @ -40 °C (cP)	60000 max	17500	12900
Yield stress	No	No	No
Pour Point (°C)	-	-60	-60
Noack (wt%)	-	11.7	11.2

Example 4-5

Base oils as prepared from the same feed as in Examples 1 and 2 under varying conditions were prepared. Properties are listed in Table 6. The cyclo-paraffins and normal and iso-paraffins of the base oil of Example 5  
 10 (see Table 6) were further analysed. In Figure 1 the content of the components, normal and iso-paraffins, 1-ring cyclo-paraffins, 2-ring cyclo-paraffins, etc. in the saturates phase as a function of their respective  
 15 carbon numbers are shown of the base oil of Example 5.

Table 6 (cont'd)

Base oil type	Example 4	Example 5	Base oil as obtained in Example 2 of EP-A-776959	Base oil as obtained by catalytic dewaxing a Shell MDS Waxy Raffinate over a Pt/synthetic ferrierite catalyst (*)	Base oil as obtained by catalytic dewaxing a Shell MDS Waxy Raffinate over a Pt/synthetic ferrierite catalyst (**)
2-ring cyclo-paraffins	1.4	1.4	0.2	0.5	0.7
3 and higher number rings cyclo-paraffins	1.2	0.3	0.2	0.7	1.1

(\*) Reaction conditions: total pressure 40 bars, WHSV=1 kg/l/h, gas recycle rate = 700 NL/kg feed and temperature of 290 °C.

(\*\*) as in (\*) but at 320 °C dewaxing temperature.

8. Lubricant composition according to any one of claims 1-7, wherein the base oil is obtainable from a process comprising the following steps:

- 5 (a) hydrocracking/hydroisomerisating a Fischer-Tropsch product having a weight ratio of compounds having at least 60 or more carbon atoms and compounds having at least 30 carbon atoms of at least 0.2 and wherein at least 30 wt% of compounds in the Fischer-Tropsch product have at least 30 carbon atoms,
- 10 (b) separating the product of step (a) into at least one or more fuel fractions and a base oil precursor fraction, and
- (c) performing a catalytic dewaxing step to the base oil precursor fraction obtained in step (b).
- 15 9. Lubricant composition according to claim 8, wherein the Fischer-Tropsch product used in step (a) has at least 50 wt%, and more preferably at least 55 wt% of compounds having at least 30 carbon atoms and wherein the weight ratio of compounds having at least 60 or more carbon
- 20 atoms and compounds having at least 30 carbon atoms of the Fischer-Tropsch product is at least 0.4 and wherein the Fischer-Tropsch product comprises a C<sub>20</sub><sup>+</sup> fraction having an ASF-alpha value (Anderson-Schulz-Flory chain growth factor) of at least 0.925.
- 25 10. Use of a lubricant according to any one of claims 1-9 as an OW-X passenger car motor oil or as an OW-X heavy duty diesel engine oil, where X is 20, 30 or 40.

## INTERNATIONAL SEARCH REPORT

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## A. CLASSIFICATION OF SUBJECT MATTER

IPC 7 C10M169/04 C10G45/58

According to International Patent Classification (IPC) or to both national classification and IPC

## B. FIELDS SEARCHED

Minimum documentation searched (classification system followed by classification symbols)

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Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched

Electronic data base consulted during the international search (name of data base and, where practical, search terms used)

EPO-Internal, WPI Data, PAJ

## C. DOCUMENTS CONSIDERED TO BE RELEVANT

Category *	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
X	WO 99 20720 A (MOBIL OIL CORP) 29 April 1999 (1999-04-29) cited in the application	1-4
A	claim 19 page 3, line 27 -page 4, line 3 page 6, line 4 -page 7, line 14 page 7, line 23 -page 8, line 24 page 12, line 3 - line 8 examples	8
X	US 6 165 949 A (HABEEB JACOB J ET AL) 26 December 2000 (2000-12-26)	1-4
A	claims 1,5,7,10,25,26,28 examples 1,2	8
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☒ Further documents are listed in the continuation of box C.☒ Patent family members are listed in annex.

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# INTERNATIONAL SEARCH REPORT

Information on patent family members

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